

EFFECTS OF HEATING ON MAGNETIC PROPERTIES OF METEORITIC
AND LUNAR SAMPLES: APPLICATION TO PALEOINTENSITY STUDIES

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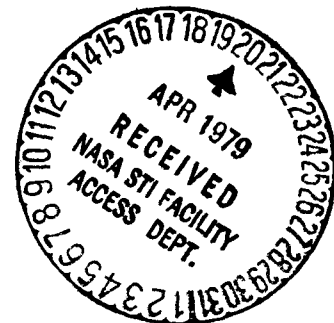
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Measurement of the magnetic properties of meteorites and lunar samples at room temperature are relatively straight-forward and unambiguous (Gose and others, 1972; Brecher and Morash, 1974; Nagata and others, 1972, 1974; Watson and others, 1974; Pearce and others, 1974; Fuller 1974; Runcorn and others, 1971; Banerjee and Hargraves, 1972; Brecher and Ranganayaki, 1975; Butler, 1972; Gus'kova, 1963; Herndon and others, 1976; Larson and others, 1973, 1974; Nagata, 1978; Nagata and Sugiura, 1976; Stacy and others, 1961; and Watson and others, 1975).

Almost all studies substantiate the fact that extraterrestrial materials generally contain an easily measurable remanent magnetization, but it is not always clear how the remanence was acquired. In the case of lunar basalts it can be argued that the magnetism is a TRM acquired during initial cooling. However, because nearly all lunar samples are fragments of preexisting rock units disaggregated catastrophically during a meteorite impact event, there exists the real possibility that the original remanence has been modified or largely overprinted during shock events (Fuller, 1974). Brecher (1976) and Brecher and others (1975) have gone so far as to say that all lunar samples have been overprinted to some degree by shock remanent magnetization (SRM). On the other hand, severe shocking can lead to momentary elevation of the rock to temperatures above the Curie points of their contained magnetic phases and can, therefore, lead to TRM or pTRM acquisition during subsequent cooling. Severely shocked lunar breccia, therefore may contain a relatively simple TRM.

If shocking or near-surface heating leads to chemical change in the rock there is always the possibility of the development of chemical remanent magnetization (CRM). In some carbonaceous chondrites it is clear that some of the magnetite has grown by some sort of gas-phase crystallization at low temperature and probably, in the process, acquired CRM. Many carbonaceous chondrites have never been subjected to elevated temperatures and it must be assumed that their remanence must, represent some sort of DRM (Nagata, 1961), IRM, CRM or a combination thereof.

In spite of the many possible mechanisms for remanence acquisition in extraterrestrial materials, most investigators are of the opinion that the remanence in some samples, particularly the high-coercivity portion, is a true TRM. This is based primarily on the character of decay during alternating

field (a.f.) demagnetization and the coercivity spectrum. If that is so, then these samples have the potential of providing data from which estimates can be made of the intensity of the ancient magnetic field in which the rocks acquired their magnetism. In many cases, the time of TRM acquisition will correspond to the time of rock origin which can be radiometrically dated.

Valid paleointensity figures, if available, are crucial to the formulation of viable theories concerning the early development of the nebular, solar, and earth-moon systems. Undoubtedly, such estimates represent the most significant magnetic information that can be obtained from extraterrestrial rocks.

Based on the acceptance of TRM in extraterrestrial samples, many workers, using meteorite and lunar samples, have made paleointensity estimates (Collinson and others, 1973; Gose and others, 1973; Banerjee and Mellema, 1974a, 1974b; Collinson and others, 1975; Fuller, 1974; Stephenson and Collinson, 1974; Stephenson and others, 1977; Cisowski and others, 1977; Sugiura and others, 1978; Banerjee and Hargraves, 1972; Butler, 1972; Nagata, 1929; Brecher, 1972; Banerjee and Mellema, 1972; Stacey and others, 1961; Brecher, 1977; Nagata and Sugiura, 1979, Weavery, 1962; Gus'kova, 1963; Gorshkov and others, 1975; Brecher and Ranganayaki, 1975).

For ordinary chondrites, estimates generally range between 0.1 and 0.4 Oe; for achondrites around 0.1 Oe; and for carbonaceous chondrites near 1 Oe. Iron meteorites, for several reasons, appear to be unusable for paleointensity analysis. Nagata (1979) concludes that the postulated values represent likely magnetic fields when the meteoritic material formed about 4.5×10^9 years ago. In the case of the achondrites the field may have been the result of a self-generating dynamo in the metallic core of a primordial planet. In the case of the other meteorites, although somewhat problematical, he suggests that the field may have stemmed from the presence of a primordial solar wind.

Paleointensity estimates of lunar samples range from about 0.015 to 1.5 Oe. In some cases investigation of sub-samples from the same parent sample, by two different research groups, has resulted in greatly divergent estimates.

To date, estimation of paleointensities, both of meteorites and lunar rocks, has principally resulted from application of 1) some form of the Thellier method (Thellier and Thellier, 1959), 2) ARM methods (Banerjee and Mellema, 1974a; Stephenson and Collinson, 1974; Collinson and others, 1973) or

3) NRM vs. IRM (Fuller, 1974). If the remanence is truly a TRM, it would seem reasonable that the Thellier-type methods, involving heating only, would be the most advantageous. Actually, only this technique has been tested through application to historic lava flows (Coe and Grommé, 1973; Khodair and Coe, 1975; Coe and others, 1978, Watson, personal communication, 1977). It is clear from the results that the method does not always work, and, moreover, the reasons for its failure are not always simple to determine. In most cases, some type of chemical alteration (for example, unmixing of magnetic phases, oxidation, reduction, reequilibration of phases) seemed to occur during the various heating and cooling steps to which the sample was subjected.

It was in response to these problems that the ARM method was devised. It has potential utility but has thus far never really been meaningfully tested. Moreover, some critics have raised serious questions concerning the fundamental validity of the method (Levi, 1974; Dunlop and others, 1975) and even some of the people using the method (Collinson and others, 1975) have commented on some of its uncertainties. Moreover, at least one heating step--to high temperatures--is necessary during application of the method.

The method of comparing NRM vs. saturation IRM circumvents some of the problems of the previous two techniques but is capable of providing only a rough order-of-magnitude estimate.

Because the Thellier-type methods have been tested and have been shown to be applicable to some terrestrial rocks, if deleterious effects during heating can be minimized, most paleomagnetists tend to favor this method and put more trust in the results. Unfortunately, meteoritic and lunar samples do not lend themselves readily to the repeated thermal cycling which is necessary with the Thellier technique.

It was in an effort to find ways of minimizing such deleterious thermal effects that we began our investigation under NASA sponsorship in 1971. During the course of our efforts we worked both with meteorites (primarily carbonaceous chondrites) and lunar samples. The work with meteorites was mostly aimed at establishing their opaque mineralogy and developing means of decreasing the adverse effects due to heating, with the hope that these techniques could be applied to thermal experiments involving lunar samples.

Meteorites

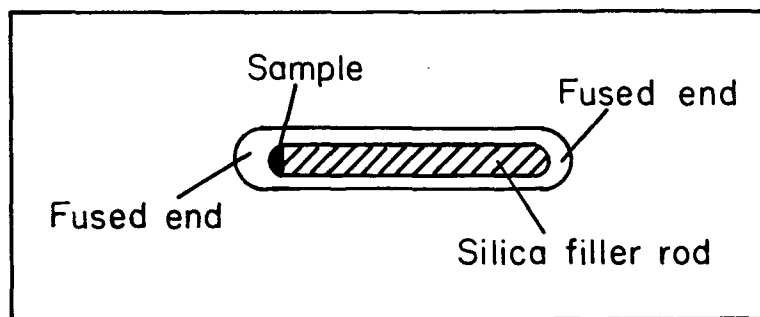
In all, we thermomagnetically examined 41 primitive meteorites of classes C1, C2, C3 and C4 chondrites (Larson and others, 1974; Watson and others, 1975; Herndon and others, 1976) by means of a modified Cahn microbalance (Larson and others, 1975). To reduce the alteration of magnetite iron and nickel-iron grains during measurement of changes in saturation magnetization with temperature (J_s-T), we flowed a mixture of H_2 and CO_2 (in an N_2 carrier gas) past the sample. The particular oxygen fugacity (fO_2) of the gas mixture could be varied in real time by changing the proportions of H_2/CO_2 . The fO_2 could be monitored during the experiment by measuring the voltage put out by an yttria-doped zirconia tube placed adjacent to the sample, a method previously developed by high-temperature experimental chemists (Sato, 1971; Nafziger and others, 1971). For safety reasons we did not use pure H_2 , but mixed it with N_2 , never permitting a mixture richer than 21% H_2 . A flow of 21% H_2 /79% N_2 was adequate for maintaining iron, nickel-iron, and magnetite in a reducing atmosphere only at temperatures above 300°C. Fortunately, below that temperature reaction rates are relatively slow, except for finely divided material. We were able to demonstrate that this system is far superior to a vacuum for controlling oxidation or reduction during heating of a sample.

In addition, we similarly constructed fugacity-controlled furnaces for use in heating and cooling samples in a zero or known field (Watson and others, 1974). Since the time of our initial use of fugacity controlled systems, several other paleomagnetists have followed suit. There now are units at Pittsburg University (Schmidt), University of Wyoming (Shive), and Toronto University (Strangway).

It became apparent, however, through our work on meteorites and synthesized samples, that gas-mixing at one atmosphere pressure is ineffective in controlling the breakdown of high-vapor-pressure constituents like troilite (FeS). During heating, troilite, particularly if finely divided, dissociates; sulfur leaves the system and iron (or iron oxide) forms (Larson and others, 1974; Watson and others, 1975; and Herndon and others, 1976). Breakdown can begin just above 100°C if the troilite grain size is very small. That heating can bring about the formation of additional ferromagnetic material was suspected soon after investigation of the first returned lunar rocks when it

was found that lunar breccia and soils contain 3 to 4 times the amount of iron found in lunar basalts (Fuller, 1974). Houslay and others (1973) and Pearce and others (1972) considered the iron to result from reduction of iron-rich silicate glass formed during meteorite impact. In light of our results, it would seem that iron formation through breakdown of troilite during and following a shock event is a process just as or even more likely than that of reduction of silicate glass.

Our solution to inhibiting FeS dissociation during heating involved encapsulation of the sample in a sealed vessel where pressures developed during the experiment would be sufficient to retard boil off of sulfur. Sealed platinum and gold tubes were found to be difficult to fashion and largely ineffective. Evaporation of several layers of gold on the samples also proved to be inadequate inasmuch as small holes in the coating always persisted. Finally, we tried encapsulation of the sample in a small evacuated silica tube in which, after the sample is introduced, a solid silica rod is fitted into the central tube void, thereby further reducing the volume of the sample chamber (See Figure 1).



Sample encapsulation in
silica tube

This method when used in conjunction with gas mixing appears to be a step in the right direction. Alteration of powdered meteoritic troilite, troilite-rich carbonaceous chondrites (for example, Allende) and selected lunar samples have been essentially eliminated by application of this procedure prior to J_s -T analysis. Interestingly enough, even though the tube is closed, it is possible for the gas environment surrounding the tube to affect the oxygen fugacity. For example, a lunar sample previously heated in air to 200°C during measurement of its dielectric properties was encapsulated and examined by J_s -T analysis. A considerable component of magnetite

($T_c=580^\circ\text{C}$) was at first noticeable in the analysis. Flowage of 21% H_2 /79% N_2 only, reduced the Fe_3O_4 to iron. Subsequently, the gas mixture was relatively enriched in CO_2 and magnetite again became detectable.

A modification of this encapsulation system is now being used in paleointensity experimentation at Toronto University.

By means of J_s -T analysis of encapsulated and unencapsulated samples in a controlled gas-buffered atmosphere we were able to determine the major magnetic and potentially magnetic constituents in all 41 carbonaceous chondrites we obtained. Moreover, by calibration of the balance, we were able to accurately estimate the magnetic saturation moments and subsequently the weight percentages of the magnetic constituents.

Interestingly enough, all C1 chondrites contain only pure magnetite (Fe_3O_4) in amounts that vary from about 8 to 11 wt%. C2 chondrites are variable--some contain only magnetite, some magnetite and iron, and some essentially no magnetic mineral. Most, particularly those containing little ferromagnetic material, contain relatively large amounts of fine-grained troilite. C3 and C4 chondrites are also highly varied in their magnetic mineralogic makeup. Some contain only Fe_3O_4 , some Fe_3O_4 and troilite, and some Fe_3O_4 and nickel-iron (6-8%Ni).

In view of the variation in physical makeup and opaque and non opaque mineralogy it is probably safe to conclude that the origin of this meteorite group is not simple (Herndon and others, 1976). For that reason, it is suspected that paleointensity estimates, many based on thermal studies in which gas control and encapsulation were not used, are generally questionable reliability. For those samples that contain large amounts of fine-grained troilite, standard Thellier type experiments would be nearly hopeless. For these, irreversible breakdown of FeS to Fe_3O_4 or Fe begins near 100°C and progressively increases with temperature. Butler (1972) and Banerjee and Hargraves (1972) attempted to use thermal techniques on the C3 Allende meteorite but were forced to discontinue their experiments at temperatures of 150°C and 130°C , respectively, because of deleterious breakdown effects. They nonetheless made estimates of the paleointensities on the basis of the low-temperature portion of the demagnetization-remagnetization plot. Low-temperature components such as these could have had a variety of modes of remanence acquisition and therefore the paleointensity estimates are highly suspect.

According to Nagata (1979) the most reliable paleointensity estimates are derived from analysis of the extremely stable remanence in C1 chondrites. Yet, these are non equilibrium mixtures of extraterrestrial debris which has undergone minimal heating, metamorphism, and compaction. As is evident from their volatile and mineralogic makeup they appear to have undergone little or no heating above 100°C. Their remanence can hardly be termed TRM but must be some admixture of DRM, CRM and even IRM. All paleointensity studies of C1 chondrite assume that the remanence is a TRM. Likely, the results to date are essentially meaningless.

Lunar Studies

Reliable paleointensity estimates based on analysis of lunar rocks no doubt constitute the potentially most-significant information obtainable by the paleomagnetic community. However, lunar samples, like meteorites, are very difficult rocks to work with during thermal experiments. They contain variable proportions of iron, nickel-iron, troilite, ilmenite, chromium ulvo spinel, and titanium chromite, in an unknown state of equilibrium. If deleterious effects during heating are to be avoided it is now clear that they must be encapsulated and subsequently heated in a gas-buffered furnace. Unfortunately many (probably most) of the published paleointensity estimates are based on studies in which alteration safeguards were minimal: samples were simply heated in air or in vacuum. It is no wonder that the intensity estimates are so contradictory.

We have examined a few of the samples used during the early era of paleointensity measurements by the Strangway and Runcorn groups (Larson, 1978). We initially thought that degraded samples such as these could serve as natural analogues by means of which we could evaluate the most advantageous way of thermally treating samples. We found, by means of petrographic observation, that the degraded samples had been variable but extensively affected by oxidation. Magnetite and even hematite (Fe_2O_3) were common and troilite has suffered breakdown. Even ulvospinel showed oxidational effects. Because of the obvious thermal degradation of these samples, any paleointensity estimates derived from them must be viewed with suspicion.

To test the utility of more sophisticated techniques we encapsulated some of the degraded material and subjected it to J_s -T analysis. Heating to high temperatures (800°C) brought about irreversible changes in the saturation magnetization curves. Some iron apparently alloyed with available elements to produce minerals of highly unusual Curie temperatures and saturation characteristics. Thermal recycling brought about a requilibration after which no further changes occurred. The results of these experiments suggest that thermally degraded samples are not suitable for meaningful paleointensity reexperimentation. They cannot serve as lunar analogues and are, therefore, largely lost to the magnetic community.

It is hoped that in the future, if lunar samples are to be heated, safeguards such as gas mixing and encapsulation will be used, such as is now being done at Toronto University under the guidance of Dr. D. Strangway. Sugiura and others (1978) have reported some of the new attempts at Toronto to estimate the paleolunar field. In these studies they used only gas-mixing fugacity control. Interestingly enough, none of five lunar samples could be shown to contain a reliable high-temperature remanence component and they were unable to make any paleofield estimates. The remanence in several samples disappeared at about 400°C or less and they conclude that this low-temperature component may have been acquired during impact events.

Presently, the Toronto group is additionally using encapsulation and it will be interesting to see what their new results will be.

Possible, at some time in the future, we will be able to perform thermal experiments without overly degrading the sample. At that time, we will have to face the problem of how the lunar rocks acquired their magnetization. for all paleointensity techniques are based on the fundamental assumption that the remanence is solely a TRM. If that is not so, and there are many reasons to question the assumption, then we will have fought hard for a technique that will produce meaningless results.

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